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IS 4528 (1977): 4, 4'-dinitrostilbene-2, 2'-disulphonic Acid (Disodium Salt) [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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IS : 4528 - 1977

*Indian Standard*  
SPECIFICATION FOR  
4, 4'-DINITROSTILBENE-2, 2'-DISULPHONIC  
ACID ( DISODIUM SALT )  
( *First Revision* )

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INDIAN STANDARDS INSTITUTION  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

December 1977

Part 2 of Gr 2

# Indian Standard

## SPECIFICATION FOR

### 4, 4'-DINITROSTILBENE-2, 2'-DISULPHONIC ACID (DISODIUM SALT)

### ( First Revision )

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**IS : 4528 - 1977**

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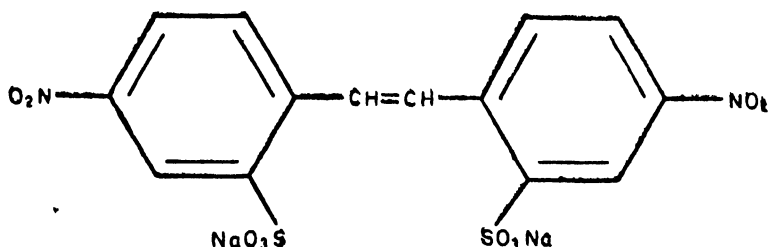
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**Indian Standard**  
**SPECIFICATION FOR**  
**4, 4'-DINITROSTILBENE-2, 2'-DISULPHONIC**  
**ACID ( DISODIUM SALT )**  
*( First Revision )*

## 0. FOREWORD

**0.1** This Indian Standard ( First Revision ) was adopted by the Indian Standards Institution on 27 July 1977, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

**0.2** 4, 4'-Dinitrostilbene-2, 2'-disulphonic acid as its disodium salt (  $C_{14}H_8O_{10}N_2S_2Na_2$  ) is an intermediate widely used in the manufacture of dyes as well as for the manufacture of diaminostilbene disulphonic acid. It is represented by the following structural formula:



**4, 4'-DINITROSTILBENE-2, 2'-DISULPHONIC ACID ( DISODIUM SALT )**  
 ( Molecular Mass 474.4 )

**0.3** This standard was first issued in 1968. It was felt by the Sectional Committee responsible for the preparation of this standard that it should be revised in the light of experience gained by the industry in recent years. In the first revision, limit of impurity, 4, 4'-dinitrodibenzyl-2, 2'-disulphonic acid has been incorporated. The assay of disodium salt is only by reduction method whereas in the original standard the material had to be assayed by two different methods.

**0.4** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960\*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## 1. SCOPE

**1.1** This standard prescribes the requirements and the methods of sampling and test for disodium salt of 4, 4'-dinitrostilbene-2, 2'-disulphonic acid.

## 2. REQUIREMENTS

**2.1 Description** — The material shall be in the form of yellowish orange paste or dry powder.

**2.2** The material shall also comply with the requirements given in Table 1.

**TABLE 1 REQUIREMENTS FOR 4, 4'-DINITROSTILBENE-2, 2'-DISULPHONIC ACID (DISODIUM SALT)**

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL NO. IN APPENDIX A
(1)	(2)	(3)	(4)
i)	Assay (on dry basis), percent by mass, <i>Min</i>	92	A-2
ii)	Matter insoluble in water (on dry basis), percent by mass, <i>Max</i>	0.1	A-3
iii)	4, 4'-Dinitrodibenzyl-2, 2'-disulphonic acid (on dry basis), percent by mass, <i>Max</i>	2.0	A-4

## 3. PACKING AND MARKING

**3.1 Packing** — The material shall be packed in steel drums (see IS : 2552-1970†) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

\*Rules for rounding off numerical values (*revised*).

†Specification for steel drums (galvanized and ungalvanized) (*first revision*).



**3.2 Marking** — Each container shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Batch number; and
- d) Net, gross and tare mass.

**3.2.1** Each container shall, in addition, be marked with label as given in Fig. 11 of IS : 1260 ( Part I )-1973\* which shall be printed with the minimum cautionary notice worded as under:

‘ POISON ! KEEP WELL CLOSED, HANDLE WITH CARE ’.

**3.2.2** The containers may also be marked with the ISI Certification Mark.

**NOTE** — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution ( Certification Marks ) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

## 4. SAMPLING

**4.1** Representative samples of the material shall be drawn as prescribed in 3 of IS : 5299-1969†.

### 4.2 Number of Tests

**4.2.1** Test for assay shall be conducted on each of the individual samples.

**4.2.2** Tests for remaining characteristics, namely, matter insoluble in water and 4, 4'-dinitrodibenzyl-2, 2'-disulphonic acid shall be conducted on the composite sample.

### 4.3 Criteria for Conformity

**4.3.1 For Individual Samples** — The lot shall be declared as conforming to the requirement of assay if each of the individual test results satisfies the relevant requirement given in Table 1.

\*Pictorial markings for handling and labelling of goods: Part I Dangerous goods (*first revision*).

†Methods of sampling and tests for dye intermediates.

**4.3.2 For Composite Samples**— For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample ( *see* 4.2.2 ), the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

## 5. TEST METHODS

**5.1** Tests shall be conducted according to the methods prescribed in Appendix A as indicated in col 4 of Table 1.

**5.2 Quality of Reagents**— Unless specified otherwise, pure chemicals and distilled water ( *see* IS : 1070-1977\* ) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

## APPENDIX A

( Table 1, and Clause 5.1 )

### METHODS OF TEST FOR 4, 4'-DINITROSTILBENE-2, 2'-DISULPHONIC ACID ( DISODIUM SALT )

#### A-1. PREPARED SAMPLE

**A-1.1** Dry the material at  $105 \pm 1^{\circ}\text{C}$  to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this *prepared sample* for tests.

#### A-2. ASSAY

**A-2.1** Carry out the assay as prescribed in 14 of IS : 5299-1969†. For reduction, follow the boiling method as prescribed in 14.3.3 with the following change:

Add 30 ml of 20 percent ( *m/v* ) sodium acetate solution before adding 50 ml of titanous trichloride solution.

#### A-3. DETERMINATION OF MATTER INSOLUBLE IN WATER

**A-3.1 Procedure**— Weigh accurately about 10 g of the *prepared sample* ( *see* A-1.1 ). Transfer this to a 500-ml beaker with 250 ml of water. Heat to 60 to  $70^{\circ}\text{C}$  with constant stirring and allow to settle. Filter through a

\*Specification for water for general laboratory use ( *second revision* ).

†Methods of sampling and tests for dye intermediates.

tared sintered or Gooch crucible, wash thoroughly and dry in an oven at  $100 \pm 2^\circ\text{C}$ . Cool in a desiccator and weigh. Repeat the process of drying and weighing until constant mass is obtained. Express this mass as a percentage of the sample taken for test and report as insoluble matter.

#### A-4. DETERMINATION OF 4, 4'-DINITRODIBENZYL-2, 2'-DISULPHONIC ACID

**A-4.0 Outline of the Method**—Aniline forms a complex with dinitrostilbene disulphonic acid, while dinitrodibenzyl disulphonic acid does not, which results in the difference in  $R_f$  values when they are allowed to run on chromatographic paper. Dinitrostilbene disulphonic acid gives two distinct spots which are due to *cis-trans* isomers.

##### A-4.1 Apparatus

**A-4.1.1 Paper Chromatographic Chamber**

**A-4.1.2 Micro Pipettes**—10 microlitre capacity.

**A-4.1.3 Spray Bottle**

##### A-4.2 Reagents

**A-4.2.1 4, 4'-Dinitrostilbene-2, 2'-Disulphonic Acid**—Free from 4, 4'-dinitrodibenzyl-2, 2'-disulphonic acid.

**A-4.2.2 4, 4'-Dinitrodibenzyl-2, 2'-Disulphonic Acid Solution**—0.5 percent ( $m/v$ ). Dissolve 0.5 g of dinitrodibenzyl disulphonic acid in 8 ml of 1 N sodium hydroxide solution and make the volume to 100 ml with dimethyl formamide.

**A-4.2.3 Aniline Acetate Solution**—For preparing 5 litres of this solution mix 46.5 ml of aniline and 212.5 ml of glacial acetic acid in 5-litre volumetric flask and dilute with water up to the mark.

**A-4.2.4 Methanolic Potassium Hydroxide Solution**—1 N.

**A-4.2.5 Whatman Chromatographic Paper No. 1**—or equivalent.

**A-4.2.6 Sodium Hydroxide Solution**—1 N.

##### A-4.3 Procedure

**A-4.3.1** Weigh accurately 5.0 g of the prepared sample (*see* A-1.1), dissolve in 8 ml of sodium hydroxide solution and make the volume to 100 ml with dimethyl formamide in a 100-ml volumetric flask. Similarly weigh 5.0 g of the pure 4, 4'-dinitrostilbene-2, 2'-disulphonic acid, add 10 ml of 0.5 percent ( $m/v$ ) solution of dinitrodibenzyl disulphonic acid solution and treat in the same way as sample.

**A-4.3.2** Spot 5 microlitre each of the sample solution and reference solution on Whatman No. 1 or an equivalent paper. Suspend the paper in the chamber and keep the aniline acetate solution tank at 20 to 25°C. Allow the solvent to ascend up to 30 cm which takes about 5 to 6 hours. The temperature of the chamber shall be 20 to 25°C.

**A-4.3.3** Take out the paper and dry at room temperature. Pass it through a trough containing methanolic potassium hydroxide. Dry in an air oven at 100 to 120°C. Violet coloured spots are developed.

**A-4.4 Results** — Different spots obtained shall be as given below:

<i>Substance</i>	<i>Zone</i>	<i>Rf value</i>	<i>Colour</i>
Dinitrostilbene disulphonic acid	I	0.53	Violet
Dinitrodibenzyl disulphonic acid	II	0.63	Violet
Dinitrostilbene disulphonic acid	III	0.76	Violet

# INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

## Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

## Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

## Derived Units

Quantity	Unit	Symbol	Conversion
Force	newton	N	1 N = 1 kg.1 m/s <sup>2</sup>
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m <sup>2</sup>
Frequency	hertz	Hz	1 Hz = 1 c/s (s <sup>-1</sup> )
Electric conductance	siemens	S	1 S = 1 A/V
Pressure, stress	pascal	Pa	1 Pa = 1 N/m <sup>2</sup>

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AMENDMENT NO. 1 SEPTEMBER 1986

TO

IS: 4528-1977 SPECIFICATION FOR  
4,4'-DINITROSTILBENE-2,2'-DISULPHONIC  
ACID (DISODIUM SALT)

*(First Revision)*

[Page 4, Table 1, Sl No. (iii), col 3] - Substitute  
'1.0' for '2.0'.

(FDC 11)

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